

TITLE**TWO-COAT ELECTROCOATING PROCESS****BACKGROUND OF THE INVENTION**

5 The present invention relates to a two-coat electrocoating process. More particularly, the present invention preferably relates to a process wherein a substrate is first coated via a cathodic electrodeposition system and a second coat is subsequently applied via an anodic electrodeposition system.

10 The coating of electrically conductive substrates using an electrodeposition process (also referred to as electrocoating, E-coating, Elpo and electrophoretic coating) is well known in the art. Generally, there are two known types of electrodeposition processes, anodic
15 electrodeposition and cathodic electrodeposition, both of which are performed by inducing an electrical charge to a coating composition in an immersion bath. The charged coating composition, under the influence of an electrical field, migrates to the surface of the substrate. Once the coating composition is deposited upon the substrate, the coating composition becomes insoluble and remains on the surface of the
20 substrate. The coating composition is drawn to the substrate wherein a continuous film is deposited over the surfaces of the substrate until the film of coating composition reaches a desired thickness. Typically, when a substrate is to be coated, the process comprises the steps of cleaning the surface of the substrate, converting the surface of the substrate,
25 electrocoating the substrate by applying a first coating which typically further includes a pigment whose conductivity is activated via the baking step thereby allowing for the application of the second coat, rinsing the substrate using water, baking the substrate in order to cure the first coat using an oven maintained at sufficient temperatures to produce a cross-linked finish on the substrate, applying a second coating via
30 electrodeposition, a second rinsing of the substrate, and a final baking in order to cure the second coat.

 A coating method is described in European Patent 421,247, issued to Illinois Tool Works Inc., which discloses an electrodeposition process
35 of coating metallic substrates in bulk quantities, wherein the process begins by anodically electrodepositing a film, such that the anodic primer

coat requires curing in order to prevent "touch point" or breaks in the coating. In addition, the method disclosed in '247 is intended for use only with electrically conductive substrates.

5 A two-coat electrocoating process is also described in United States Patent 5,507,928 (Bohmert et al) however, this describes only the use of cathodic or anodic compositions in its process, but does not disclose a process wherein a substrate is alternatively coated with cathodic and anodic compositions. Bohmert et al indicate that the same composition must be utilized throughout the process.

10 United States Patent 6,231,738 discloses a method of making a multilayer electrodeposited composition, wherein the first coat is a conductive coating containing at least one material which can impart conductivity to the first coating composition upon exposure to an effective amount of energy. The first coating composition is overcoated with a
15 second coating composition only after the first coating composition is subjected to said amount of energy effective to cause the first coat to become conductive.

Other electrocoat patents include U.S. Published Patent Application 2002011412 A; U.S. Patent 5,908,667 A; U.S. Patent
20 5,203,975 A; U.S. Patent 4,882,090; U.S. Patent 6,248,225; U.S. Patent 6,162,339; U.S. Patent 5,882,734; EP 426328 B; EP 1067218 A; WO 01/49794 A; WO 01/49795 A; WO 01/77239 A, however, each of these patents utilizes a conductive pigment in the first coat, and require baking of the first coat.

25 The two-coat electrocoating process of the present invention addresses several problems of those processes currently utilized within the art, such as, below normal throwpower of the first coat, multiple baking steps, and the high levels of volatile organic compound (VOC) produced by existing electrocoating and primer systems.

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SUMMARY OF THE INVENTION

The present invention relates to a process of electrocoating a substrate with at least two electrodepositable layers comprising the steps of:

- (a) cathodically electrodepositing a primer coating composition to a substrate by immersion of a substrate in a cathodic electrocoating composition;
- 5 (b) anodically electrodepositing a primer surfacer coating composition onto the substrate by immersion of the substrate in an anodic electrocoating composition; and
- (c) curing the at least two electrodepositable layers disposed upon the substrate simultaneously in a single bake;

10 wherein the primer coating composition contemplated by the inventive process is still wet when step (b) is performed. The wetness of the primer composition indicates that it is not completely sealed by high amounts of pigment and/or there is no cross-linking occurring.

DETAILED DESCRIPTION

15 The present invention provides several advantages in that it allows for good throwpower without compromising overall corrosion protection. Furthermore, the present invention provides advancements in the electrocoating industry as well as advantages to those businesses utilizing electrocoating technology because it provides production cost savings and

20 is adaptable to current plant configurations. Still further, the present invention provides environmental advantages because it may be implemented in place of less efficient applicators, for example a spray primer surfacer, thereby providing for a reduction in waste and therefore, the utilization of lower amounts of organic solvents. Additionally, the one

25 bake system of the present invention reduces energy usage, when compared to dual baking systems, which translates into the reduction of carbon monoxide levels generated due to multiple baking steps.

The present invention relates to a process for depositing multiple layers of the coating compositions upon a substrate, wherein the multiple

30 coating layers comprise at least a primer coating composition and a primer surfacer coating composition. The primer provides corrosion protection while the primer surfacer, which is subsequently applied onto the primer, provides weatherability as well as protection of the primer. Application of the primer and primer surfacer layers may be performed via any

35 combination of anodic and cathodic deposition systems. However, application of the primer preferably occurs via a cathodic system, whereas

the application of the primer surfacer preferably occurs via an anodic system.

Prior to the application of the primer coating composition, the substrate is initially prepared wherein the substrate's surface is cleaned and converted, thereby allowing it to receive the electrocoating compositions. The metal substrate can be cleaned according to those conventional methods known in the art including, but not limited to, an alkali wash with specialized surfactant additives to remove accumulated oils, grease, adhesives, excess sealer, lubricants and dirt. Further preparation of the substrate for receiving an electrocoat includes conversion, a process generally comprising the steps of preconditioning the substrate using sodium or titanium nitrate; phosphating the substrate which may be performed using any of the well known pretreatment techniques utilized in applying a coating comprising iron phosphate, zinc phosphate, zinc-iron phosphate, wherein zinc phosphate is preferred; and sealing the phosphate coating. Typically, the substrate is rinsed at least once in order to passivate the deposited conversion coating and neutralize any remaining water-soluble salts, thereby allowing for proper electrodeposition to occur.

More specifically, the present invention relates to a process of electrocoating a substrate with at least two electrodepositable layers comprising the steps of:

- (a) cathodically electrodepositing a primer coating composition to a substrate by immersion of a substrate in a cathodic electrocoating composition;
 - (b) anodically electrodepositing a primer surfacer coating composition onto the substrate by immersion of the substrate in an anodic electrocoating composition; and
 - (c) curing the at least two electrodepositable layers disposed upon the substrate simultaneously in a single bake;
- wherein the primer coating composition contemplated by the inventive process is still wet when step (b) is performed. The wetness of the primer composition indicates that it is not completely sealed by high amounts of pigment and/or there is no cross-linking occurring.

The process of the present invention allows for the primer layer to retain moisture, which is trapped in the primer layer, which is then

electrolyzed resulting in the generation of H ions to be available for the deposition of the primer surfacer layer.

According to the process of the present invention, the substrate may be of a metallic nature, such as, aluminum or a non-metallic nature, such as, a rigid or flexible plastic. Preferably the substrate is an electroconductive metal such as aluminum or steel, including galvanized steel, cold-rolled steel, zinc-coated steel, zinc-iron coated steel, and nickel-zinc coated steel. Conductivity of the substrate is necessary and must either inherently exist in the substrate or must be imparted to the substrate via using any conventionally known electrically conductive imparting or other appropriate method. Those practitioners skilled in the art recognize and understand the steps necessary to impart the required conductivity to a substrate of a non-metallic nature and therefore such procedures are not reproduced herein.

Application of the primer coating composition is preferably via cathodic electrodeposition. It is preferred because during anodic electrodeposition, the substrate acts as the anode receiving the direct electric current. Electrochemical dissolution of the conversion coating and/or the base metal substrate occurs and the subsequent inclusion of these dissolve metal ions in the deposited paint film can have several side effects including reduced corrosion resistance and non-uniform staining of the coating due to external oxidation of the dissolved metal ions.

Typically, cathodic electrocoating occurs via immersion of a substrate in an electrocoating bath where generally, the bath has a temperature in the range of about 27°C (80°F) to about 43°C (110°F), however a bath temperature of about 35°C (95°F) is preferred. The immersion time of the substrate generally ranges from about 1 minute to about 5 minutes, however the preferred immersion time is about 2 minutes. Generally, the voltage may range from about 1 volt up to several thousand volts, however typically, the voltage ranges from about 50 to about 500 volts using the coated substrate as the cathode, wherein a voltage of about 220 volts is preferred. Skilled practitioners in the art understand that the bath temperatures, immersion times, voltage, coating thickness and distance of the substrate from the electrode, depend not only on one another, but also vary according to the type of cathodic coating composition and substrate utilized in the electrodeposition process and those practitioners are aware of the adjustments necessary to optimize the

process. Practitioners within the art will further recognize that the thickness of the primer coating layer may be varied according to the application for which the substrate is to be used. Generally, however, in order to provide the desired level of corrosion protection, the primer
5 coating composition should have a thickness ranging from about 0.5 to about 1.2 millimeters, however a thickness of about 0.8 to about 0.9 millimeters is preferred.

Electrophoretically depositable cathodic coating compositions or primer compositions, which may be used according to the present
10 invention, include any known cathodically depositable electrocoating compositions including, but are not limited to, those disclosed in U.S. Patent 6,123,822 (Chung et al.) which is incorporated by reference herein in its entirety. Preferably, the primer composition comprises an aqueous coating composition having a solids content of about 5% to about 50% by
15 weight of an emulsion containing a cathodic film-forming resin binder, a blocked polyisocyanate cross-linking agent, optional additives, and the like. Further, the primer composition usually contains organic coalescing solvents, however, such organic solvents are present in minimal amounts.

The composition contains 0.01% to 5% by weight, based on the
20 weight of the primer composition, of an alkyl tin oxide as the catalyst for the reaction of the film-forming resin with the isocyanate. Typically, the alkyl tin oxides used are dipropyl tin oxide, diethyl tin oxide, dibutyl tin oxide and the like, wherein dibutyl tin oxide is preferred due to its catalytic activity. The alkyl tin oxide is dissolved with an organic or
25 inorganic acid. Typically useful neutralizing acids are lactic acid, acetic acid, formic acid, sulfamic acid, alkane sulfonic acids, such as, methane sulfonic acid, ethane sulfonic acid and propane sulfonic acid, resulting in the primer coating composition having a pH of about 5 to about 8.

Another aspect of the present invention relates to primers
30 containing pigment dispersions or pastes as are known within the art, as well as the use of primer compositions that are essentially free of pigmentation.

The term "essentially free", as used herein, refers to a coating composition wherein the level of pigments present in the composition is
35 below detectable levels such that the coating composition is clear.

The process of the present invention preferably utilizes a clear non-pigment containing primer coating composition. Processes utilizing a pigment-free primer are preferred because such primer compositions have greater stability during electrodeposition since there are no pigments to settle out. Additionally, pigment-free primers are much easier to manufacture since pigment dispersions are not a required component. Elimination of the use of pigment dispersions provides substantial savings in both time and materials. While the coatings contemplated by the present invention include a pigment paste or dispersion, such as those which are well known in the art, coatings containing less than about 5% pigmentation are preferred. More preferably, the present invention provides for the use of coating compositions containing an amount of pigment that is below detectable levels. Generally in the art, the presence of at least one conductive pigment in the primer requires a low coating voltage resulting in poor throwpower. However, in the present invention, due to the lack of pigment or reduced pigment level in the primer, normal coating voltages, rather than reduced coating voltages, are utilized resulting in normal throwpower.

"Throwpower" as used herein, shall refer to a measure of the effectiveness of an electrodepositing coating at penetrating and coating recessed or interior areas of a substrate. Electrodeposition of coating follows the force lines of the electrical field that exist in an electrocoating bath between the anode and the cathode. These force lines diminish as they penetrate into the recessed areas of the substrate and cease to exist when the recessed area is too deep and a coating will not be deposited in such an area. Good throwpower is an important aspect in order to maximize an electrocoating's anticorrosion effectiveness.

Typically, the cathodic film forming resin binder of the emulsion component used to form the cathodic electrocoating composition is an epoxy amine adduct and a blocked polyisocyanate cross-linking agent and is dispersed in an aqueous medium. The epoxy amine adduct is formed of an epoxy resin which preferably is chain extended and then reacted with an amine. Examples of cathodic film-forming resins include, but are not limited to those described in U.S. Patent 6,123,822 (Chung et al); U.S. Patent 5,070,149 (DeBroy et al); U.S. Patent 3,922,253 (Jerabek et al); U.S. Patent 4,419,467 (Wisner et al); U.S. Patent 4,137,140 (Belanger);

U.S. Patent 4,468,307 (Wisner et al); each of which is incorporated by reference herein in its entirety.

The preferred epoxy resin used in the epoxy amine adduct is polyepoxy hydroxy ether resin having an epoxy equivalent weight of 100-
5 2000. Epoxy equivalent weight is the weight of resin in grams which contains one gram equivalent of epoxy.

These epoxy resins can be any epoxy hydroxy containing polymer having a 1,2 epoxy equivalency of two or more epoxy groups per molecule. Preferred are polyglycidyl ethers of cyclic polyols, however,
10 polyglycidyl ethers of polyhydric phenols, such as, bisphenol A are even more preferred. The polyepoxides can be produced by etherification of polyhydric phenols with epihalohydrin or dihalohydrin, such as epichlorohydrin or dichlorohydrin in the presence of alkali. Examples of
15 suitable polyhydric phenols are 2,2-bis-(4-hydroxyphenol)ethane, 2-methyl-1,1-bis-(4-hydroxyphenol)propane, 2,2-bis-(4-hydroxy-3-tertiarybutylphenyl)propane, 1,1-bis-(4-hydroxyphenol)ethane, bis-(2-hydroxynaphthyl)methane and 1,5-dihydroxy-3-naphthalene.

In addition to polyhydric phenols, other cyclic polyols can be used in preparing the polyglycidyl ethers of cyclic polyol derivatives.
20 Examples of such cyclic polyols include alicyclic polyols, preferably cycloaliphatic polyols, such as 1,2-bis(hydroxymethyl)cyclohexane, 1,3-bis(hydroxymethyl)cyclohexane, 1,2-cyclohexane diol, 1,4-cyclohexane diol and hydrogenated bisphenol A.

The polyepoxides hydroxy ether resins can be chain extended with
25 any of the aforementioned polyhydric phenols, preferably bisphenol A, polyether or polyester polyols which enhance flow and coalescence. Typical useful polyol chain extenders are polycaprolactone diols such as Tone 200® series (available from Union Carbide Corporation), polyoxypropylene diamine, such as Jeffamine D-2000® having a
30 molecular weight of about 2000 (available from Texaco Chemical Company) and ethoxylated Bisphenol A such as SYNFAC 8009® (available from Milliken Chemical Company). Examples of polyether polyols and conditions for chain extension are disclosed in U.S. Patent 4,486,307, while examples of polyester polyols for chain extension are
35 disclosed in U.S. Patent 4,148,772, each of which is incorporated herein by reference in its entirety.

Ketimines can be used in the present invention with an epoxy amine adduct and are prepared from ketones and primary amines. The water formed is removed, for example, by azeotropic distillation. Suitable ketones include dialkyl, diaryl and alkylaryl ketones having 3-13 carbon atoms. More particularly, examples include acetone, methyl ethyl ketone, methyl n-butyl ketone, methyl isobutyl ketone, methyl isoamyl ketone, methyl aryl ketone, ethyl isoamyl ketone, ethyl amyl ketone, acetophenone, and benzophenone. Suitable diamines include ethylene diamine, 1,3-diaminopropane, 1,4-diaminobutane, 1,6-diaminohexane, 4,9-dioxadodecane and 1,12-diamine. A particularly useful ketimine is diketimine, which is the ketimine of diethylene triamine and methyl isobutyl ketone.

Suitable useful primary and secondary amines used to form the epoxy amine adduct include diethyl amine, methyl ethyl amine, methyl ethanol amine, ethyl ethanol amine, mono ethanol amine, ethyl amine, dimethyl amine, diethyl amine, propyl amine, dipropyl amine, isopropyl amine, diisopropyl amine, butyl amine, and dibutyl amine, however alkanol amines such as methyl ethanol amine are preferred.

Examples of suitable cross-linking agents include those disclosed in U.S. Patent 6,123,822 (Chung et al.), which is incorporated by reference herein in its entirety, and include, but are not limited to, blocked polyisocyanates, including but not limited to, aliphatic, cycloaliphatic and aromatic isocyanates, such as hexamethylene diisocyanate, cyclohexamethylene diisocyanate, toluene diisocyanate, methylene diphenyl diisocyanate, polymeric methylene diphenyl diisocyanate, and the like wherein the preferred blocked polyisocyanate cross-linking agent is methylene diphenyl diisocyanate.

Examples of suitable optional additives include ultra-violet absorbers, antioxidants, wetting agents, surfactants and defoamers and combinations thereof such as those disclosed in U.S. Patent 6,123,822 (Chung et al.), which is incorporated by reference herein in its entirety, including, but not limited to, alkyl imidazolines such as those available from Ciba-Geigy Industrial Chemicals as "Amine C", acetylenic alcohols available from Air Products and Chemicals as "Surfynol® 104". These optional ingredients, when present, constitute from about 0.1% to about 20% by weight of the binder of the coating composition.

Another optional additive are plasticizers, which are used to promote flow. Examples of suitable plasticizers include high boiling water immiscible materials, such as ethylene or propylene oxide adducts of nonyl phenols or bisphenol A. Plasticizers are typically utilized at a
5 level in the range of 0.1% to 15% by weight of the resin binder of the composition.

Application of the primer surfacer coating composition is preferably via anodic electrodeposition. The electrodeposition of the primer surfacer may be performed by immersion of the cathodic
10 composition coated substrate in an anodic electrocoating bath having a temperature in the range of about 27°C (80°F) to about 43°C (110°F), however the temperature of the anodic electrocoating bath is preferably about 35°C (95°F). The immersion period of the substrate ranges from about 1 minute to about 5 minutes, however the preferred length of time is
15 about 2 minutes. Generally, voltage requirements for the primer surfacer are lower than those for the cathodic electrodeposition of the base coat. The voltage required for the anodic electrodeposition ranges from about 75 to about 250 volts using the coated substrate as the anode; however, the voltage is preferably about 80 volts. Skilled practitioners in the art
20 understand that the bath temperatures, immersion times, voltage, coating thickness and distance of the substrate from the electrode depend not only on one another, but also vary according to the type of anodic coating composition and substrate utilized in the electrodeposition process and those practitioners are aware of the adjustments necessary to optimize the
25 process. Practitioners within the art will further recognize that the thickness of the primer surfacer coating layer may be varied according to the application for which the substrate is to be used, however, the primer surfacer preferably has a thickness of about 0.7-1.5 millimeters.

The primer surfacer composition contemplated by the inventive
30 process is an anodic waterborne system, which may be clear or pigmented, and is applied while the primer is still wet. The wetness of the primer composition indicates that it is not completely sealed by high amounts of pigment and/or there is no cross-linking occurring. Electrophoretically depositable anodic coating compositions that may be used according to the
35 present invention include any known anodically depositable electrocoating compositions. Preferably, the primer surfacer is the reaction product of polyisocyanates, an alcohol and an acid-functional alcohol, wherein the

oligomer is neutralized with an amine and subsequently combined with water, thus forming the urethane oligomer. Also, a low molecular weight composition is preferred so that the electrodeposition process will produce a sufficient film thickness when performed at a low voltage. A low
5 molecular weight system is preferred because it allows for the use of lower voltages in the electrodeposition process. The primer surfacer can have a number average molecular weight of less than about 3000, preferably ranging from about 500 to about 2000, however, more preferably the number average molecular weight ranges from about 600 to about 1200.

10 Examples of suitable anodic electrodeposition coatings include, but are not limited to, urethane oligomers, waterborne anodic polyesters, anodic acrylics and those coatings described in U.S. Patent 4,968,399 (Tsuchiya et al) and U.S. Patent 4,175,018 (Gacesa) each of which is hereby incorporated by reference herein in its entirety. The preferred low
15 molecular weight anodic primer surfacer is a urethane oligomer emulsion as described in the Examples, part B.

Electrophoretically depositable anodic coating compositions or primer compositions, which may be used according to the present invention, include any known anodically depositable electrocoating
20 compositions including, but are not limited to, those disclosed in U.S. Patent 4,968,399, which is incorporated by reference herein in its entirety. Preferably, the primer surfacer coating composition comprises an anodic film-forming resin binder, a cross-linking agent, and optional additives.

The anionic resins, which are known in the art, have an anionic
25 functional group which gives a negative charge and a hydrophilic nature to the resin. Examples of suitable groups are carboxyl groups, sulfonic acid groups, phosphate groups and the like. Preferred anionic aqueous resins are maleic natural or synthetic drying oil, a maleic polybutadiene resin, a half ester or half amide thereof, and anionic acrylic resin and the like.

30 The anionic resin may be prepared by copolymerizing a (meth)acrylate with an ethylenically unsaturated monomer having an acid group and optionally, another ethyleneically unsaturated monomer. Examples of suitable (meth)acrylates include methyl (meth)acrylate, ethyl (meth)acrylate, butyl (meth)acrylate, hexyl (meth)acrylate, 2-ethylhexyl
35 (meth)acrylate, lauryl (meth)acrylate, 2-hydroxyethyl (meth)acrylate, glycidyl (meth)acrylate and the like.

Examples of suitable ethyleneically unsaturated monomers having an acid group include (meth)acrylic acid, crotonic acid, itaconic acid, maleic anhydride, sulfoacrylate, mono(2-hydroxyethylacrylate) acid phosphate and the like. The ethyleneically unsaturated monomer which is
5 an optional component includes styrene, vinyl toluene, acrylonitrile, acrylamide, vinyl acetate and the like. Representative examples of the anionic acrylic resin include Coatax WE-804 and Coatax WE-822 (both available from Toray Industries Inc.).

Examples of suitable cross-linking agents of the present invention
10 include, but are not limited to, those described in U.S. Patent 4,968,399, which is incorporated by reference herein in its entirety, such as etherified methylol phenols, blocked isocyanates, methylolphenols, and melamine resins, and when the aminated polybutadiene contains remaining oxirane oxygen atoms, the etherified methylolphenols may be combined with
15 tetrabromobisphenol A.

Examples of suitable pigments for use in the present invention may any conventionally used or known within the art including, but not limited to, titanium dioxide, basic lead silicate, strontium chromate, iron oxide, zinc chromate, carbon black, talc, aluminum silicate, precipitated barium
20 sulfate, basic lead sulfate, aluminum phosphomolybdate, zinc hydroxy phosphite, a metallic pigment, such as zinc powder, an extender pigment, clay and combinations thereof.

The primer surfacer composition is primarily neutralized with an amine or base and then emulsified with the crosslinking agent and the
25 aqueous medium. The amines and/or bases for neutralizing the anionic aqueous resin includes ammonia, diethanolamine, triethanolamine, methylethanolamine, N,N-dimethylethanolamine, N,N-diethylethanolamine, diethylamine, triethylamine, morpholine, potassium hydroxide, 2-amino-2-methyl-1-propanol, aminoethyl propanol, and the
30 like. Typically, the anodic composition has a pH of about 6.0 to 10.0.

Although not specifically described in detail, it should be appreciated that both the cathodic and anodic electrodeposition baths should be replenished and adjusted to compensate for the coating composition which was removed from the bath. Replenishing the bath in a
35 continuous manner is well known in the art and further explanation is considered unnecessary.

After application of the coating compositions, the multi-layered coated substrate is baked in order to cure the multiple coating layers. The present invention thus provides a process for the production of multi-layered coatings by electrophoretic deposition of coating compositions onto a substrate, and the joint baking of the multiple layered coatings in a single step. The process of the present invention utilizes a single bake to cure the multiple layers of cathodic and anodic coating compositions deposited on the substrate. The baking, and thereby the curing of the electrodeposition layers, may be performed by any baking method known within the art, including, but not limited to, an infrared bake, a near infrared bake, convection ovens, radiant heat ovens, combination radiant/convection ovens, or other means for converting a wet, low molecular weight applied coating composition to a dry high molecular weight cross-linked film.

Typically the coated substrate is cured for about 1 minute to about 40 minutes at temperatures ranging from about 90°C (194°F) to about 200°C (392°F). Those skilled in the art recognize and understand that the curing time will vary according to the various cathodic and anodic coating compositions utilized in the process of the present invention.

Another aspect of the present invention relates to the reduced amounts of VOC's used during the electrodeposition process thereby resulting in reduced VOC pollution. Both the cathodic and the anodic electrodeposition systems utilize organic solvents during the application process. However, the present invention provides for reduced levels of VOC's wherein, up to about 0.3 lbs. per gallon of organic solvents are utilized. Preferably, however, the process of the present invention does not utilize any organic solvents due to its use of waterborne coatings.

The embodiments of the present invention are further defined in the following Examples. It should be understood that these Examples, while indicating preferred embodiments and the most preferred embodiments of the present invention, are given by way of illustration only. From the above discussion and these Examples, one skilled in the art can ascertain the essential characteristics of this invention, and without departing from the spirit and scope thereof, can make various changes and modifications of the invention to adapt it to various uses and conditions. Thus various modifications of the present invention in addition to those shown and described herein will be apparent to those

skilled in the art from the foregoing description. Although the invention has been described with reference to materials and embodiments, it is to be understood that the invention is not limited to the particulars disclosed, and extends to all equivalents within the scope of the claims.

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EXAMPLES

In the examples recited below, all parts and percentages are on a weight basis unless otherwise indicated.

10 The term "epoxy equivalent weight" as used herein refers to the weight of resin (in grams) which contains one gram equivalent of epoxy.

A. Preparation of the Cathodic Electrocoat System

Preparation of the Dibutyl Tin Oxide Solution

15 At room temperature, dibutyl tin oxide (DBTO) was dissolved in a mixture of 94 parts of 70% methanesulfonic acid, 47 parts of a surfactant mix (a mixture of 120 parts of alkyl imidazoline, 120 parts of acetylenic alcohol, and 120 parts butoxyethanol from Air Products and Chemical Inc.), 14 parts butanol, and 30 parts deionized water. This DBTO solution was added to 4476 parts of deionized water.

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Preparation of the Emulsion Component of the Cathodic Coating Composition

25 The following ingredients were charged into a suitable reaction vessel: 1046 parts of Epon 828[®] (glycidyl ether of bisphenol A, having an epoxy equivalent weight of 188, from Shell Chemical Company), 300 parts of bisphenol A, 376 parts ethoxylated bisphenol A, having a hydroxyl equivalent weight of 248, (SynFac 8009[®] from Milliken Company), 2 parts of dimethyl benzyl amine, and 91 parts of xylene. The resulting mixture was heated to 160°C (320°F) under a nitrogen blanket and held at this
30 temperature for 1 hour. 3 parts dimethyl benzyl amine were then added and the mixture was held at 147°C (297°F) until an epoxy equivalent weight of 1050 was obtained. 1503 parts of a blocked isocyanate cross-linker (diethylene glycol monobutyl ether and trimethylolpropane with 87.5:12.5 equivalent ratio blocked MODUR[®] MR, a polymeric methylene diphenyl

diisocyanate from Bayer, with 75% solids) were added. 113 parts of diketimine (a reaction product of diethylenetriamine and methyl isobutyl ketone at 72.7% solids) and 99 parts of methyl ethanol amine were added. The mixture was held at 120°C (248°F) for 1 hour. The resulting resin was

5 added to and emulsified in the above-prepared water containing DBTO, methanesulfonic acid, butanol, and surfactant. The resulting emulsion was stripped to remove organic solvent to give an emulsion having a solids content of 36%.

10 An electrocoating bath comprising the primer coating composition was prepared by blending the following constituents together.

Ingredients	Parts by Weight
Emulsion component of the primer coating composition	4166
Deionized Water	3333

B. Preparation for the Anodic Electrocoat System

15 Preparation of the Emulsion Component (urethane oligomer) of the Anodic Coating Composition

The following ingredients were charged into a suitably equipped reaction vessel under a nitrogen blanket: 149 parts of methyl isobutyl ketone, 363 parts of isocyanurate of hexanediisocyanate (Desmodur®3300 from Bayer), and 0.05 parts of dibutyltindilaurate. 162 parts of 2-

20 ethylhexanol were added using an addition funnel over 30 minutes were added to the reaction vessel and held at 120°C (248°F) for 45 minutes, and then cooled to 70°C (158°F). 54 parts of dimethylolpropionic acid were then added and held at 75°C (167°F) until all NCO was gone. The resin was added to a mixture of 1920 parts of deionized water and 37 parts of

25 aminomethylpropanol (95%) under agitation. The emulsion was stripped to remove organic solvent to give a urethane oligomer emulsion having a solids content of 20%.

C. Preparation of the Pigment Paste

A pigment paste was prepared by blending 208 parts of the above described urethane oligomer emulsion, 43 parts of Resimene® 747 (methylated melamine from Solutia), SynFac®8034 (alkylaryl polyether from Milliken), 361 parts of deionized water, 360 parts of titanium dioxide, and 40 parts of barium sulfate in a suitable reaction vessel. The resultant blend was ground in a suitable mill to a Hegman No. 7 particle size.

An electrocoating bath comprising the primer surfacer coating composition was prepared by blending the above constituents together.

Ingredients	Parts by Weight
Urethane oligomer emulsion	852
Resimene® 747	73
Pigment paste	128
Deionized Water	447

D. Coating via the Two-coat Electrocoat System

The electrocoat bath for the primer coat was held at about 35°C (95°F) and electrocoating was performed using 220 volts with the coated, phosphatized panel acting as the cathode. The primer-coated panel was then rinsed with deionized water. After the panel was rinsed, a second electrocoating bath, held at 32°C (90°F) with the coated panel as anode, contained a primer surfacer coating which was applied to the panel, wherein the electrocoating was performed using 80 volts. In order to cure the multiple film layers of coating compositions, the panel was then baked at 182°C (360°F) for 17 minutes. The cured films passed 100 double rubs with a methyl ethyl ketone soaked cloth which showed an excellent cure of the films. The film was smooth, wherein the smoothness was 15 microinches as measured by a Profilometer.